Eichrom Method and Application Note Updates

Daniel McAlister, Senior Chemist

eichrom®

RRMC
Radiobioassay & Radiochemical Measurements Conference

RRMC 2014
Knoxville, Tennessee USA
October 27 - October 31, 2014

Outline

- Updates to Eichrom “classic” methods

- Application Notes for new methods

- Highlights of important steps

Eichrom Methods

- No major changes to the chemistry

- Format/Organization

- Separated CeF_3/Electrodeposition into Separate Methods

- Improve Reference Material

- Add One Page Flowcharts

- Column and Cartridge Based Methods

Method	Analyte	Matrix	Column	Cartridge
ACS07	U	soil	x	x
ACU02	Am-Cm/Pu/U	urine	x	x
ACW01	U/Th	water	x	x
ACW02	U	water	x	x
ACW03	Am-Cm/Pu/U	water	x	x
ACW04	Am	water	x	x
ACW08	Th/Np	water	x	x
ACW10	Th	water	x	x
ACW11	Total Alpha	water	N/A	
ACW13	Th/Pu/U	water	x	x
ACW-16	Am-Cm/Pu/U/Np/Th	water	x	x
ACW-17	Am-Cm/Pu/U/Np/Th/Sr	water	x	x
FEW01	Fe	water	x	x
NIW01	Ni	water	x	x
OTS01	Pb	soil	x	x
OTW01	Pb/Po	water	x	x
OTW02	Tritium	water	x	x
RAW01	Ra	water	x	x
RAW03	Ra	water	x	x
RAW04	Ra	water	x	x
SPA01	CeF3 ppt		N/A	
SPA02	Electrodeposition		N/A	
SPA03	Am-Rare Earth Separation		x	x
SRW01	Sr	water	x	x
TCS01	Tc	soil	x	x
TSU01	Tc	urine	Disk	
TCW01	Tc	water	x	x
TCW02	Tc	water	Disk	
TP01	Set-up of U-232 Self-Cleaning Tracer			
VBS01	Vacuum Box System Guide			

Americium, Plutonium and Uranium in Urine (with Vacuum Box System)

1. Scope

1.1. This procedure describes a method for separation of americium, plutonium and uranium in urine. After completing this method, source preparation for measurement of americium, plutonium and uranium by alpha spectrometry is performed by electrolytic deposition onto stainless steel planchets (Eichrom Method SPA02) or by rare earth fluoride microprecipitation onto polypropylene filters (Eichrom Method SPA01).

1.2. This method does not address all aspects of safety, quality control, calibration or instrument set-up. However, enough detail is given for a trained radiochemist to achieve accurate and precise results for the analysis of the analyte(s) from the appropriate matrix, when incorporating the appropriate agency or laboratory safety, quality and laboratory control standards.

2. Summary of Method

2.1. Americium, plutonium and uranium are separated by Eichrom UTEVA and TRU resins prior to measurement by alpha spectrometry. A calcium phosphate precipitation technique can be used to concentrate and remove actinides from urine samples. Tracers are used to monitor chemical recoveries and correct results to improve precision and accuracy.

3. Significance of Use

3.1. This is a rapid, reliable method for measurement of actinides in urine samples that is more cost-effective and efficient than traditional ion exchange, solvent extraction and precipitation techniques. It also takes advantage of vacuum assisted flow and the ability to stack resin cartridges for simultaneous extraction of U, Pu and Am on UTEVA and TRU Resins. This approach can reduce resin separation time by more than half when compared to methods using gravity flow resin columns.

4. Interferences

4.1. Actinides with unresolvable alpha energies such as Am-241 and Pu-238 or Np-237 and U-234 must be chemically separated to enable measurement. This method separates these isotopes effectively.

4.2. Very high levels of phosphate may reduce the recovery of actinides during the calcium phosphate precipitation and column separation. Adjusting the amount of phosphate added to coprecipitate the actinides may be necessary in these cases.

5. Apparatus

5.1. *Analytical balance* - 0.0001 g sensitivity
5.2. *Cartridge reservoirs*-10mL Eichrom Part AC-200-RV10 or 20mL Eichrom Part AC-200-RV20
5.3. *Centrifuge, with rotor and carriers for 50mL and 250mL tubes*
5.4. *Centrifuge tubes,* 50mL and 250mL
5.5. *Eichrom vacuum box system*- Eichrom Part No. AC-12-BOX or AC-24-BOX
5.6. *Vacuum Box White Inner Support Tube-PE*- Eichrom Part No. AC-1000-TUBE-PE
5.7. *Vacuum Box Yellow Outer Tips*- Eichrom Part No. AC-1000-OT
5.8. *Fume hood*
5.9. *Hotplate*
5.10. *Vacuum Pump* - 115 V, 60 Hz Fisher part no. 01-092-25 (or equivalent) or house vacuum
5.11. *Glass stir rods*
5.12. *Vortex mixer*

6. Reagents

Note: Analytical grade or ACS grade reagents and trace metal grade (or equivalent) acids are recommended. Evaluation of key reagents, such as aluminum nitrate and ammonium hydrogen phosphate, for contribution to method background levels from naturally occurring radioactive materials is recommended.

Deionized water, All reagents are prepared with deionized water
Appropriate tracers or standards (U-232, Am-243, Pu-242 or Pu-236)
Ammonium hydroxide(57%), concentrated NH_4OH
Barium Chloride, $BaCl_2{\cdot}2H_2O$
Hydrochloric acid (37%), concentrated HCl
Hydrofluoric acid (49%), concentrated HF
Nitric acid (70%), concentrated HNO_3
Aluminum Nitrate Nonahydrate, $Al(NO_3)_3{\cdot}9H_2O$
Oxalic Acid Dihydrate, $H_2C_2O_4{\cdot}2H_2O$
Ammonium Oxalate Monohydrate, $(NH_4)_2C_2O_4{\cdot}H_2O$
Ammonium Hydrogen Phosphate, $(NH_4)_2HPO_4$
Ammonium Thiocyanate, NH_4SCN
Ascorbic Acid Powder, $C_6H_8O_6$

Calcium Nitrate, $CaNO_3$
Sodium Nitrite, $NaNO_2$
Ferric Nitrate Nonahydrate, $Fe(NO_3)_3{\cdot}9H_2O$
Sulfamic Acid, H_2NSO_3
Hydrogen Peroxide (30%), concentrated H_2O_2
1-Octanol, $C_8H_{17}OH$
TRU Resin - 2mL prepacked cartridge, 50-100µm, Eichrom Part TR-R50-S
UTEVA Resin - 2mL prepacked cartridge, 50-100µm, Eichrom Part UT-R50-

6.1. *Ammonium bioxalate (0.1M)*- Dissolve 6.3g of oxalic acid and 7.1g of ammonium oxalate in 900mL of water. Dilute to 1L with water.
6.2. *Ammonium hydrogen phosphate (3.2M)*- Dissolve 208g of $(NH_4)_2HPO_4$ in 400mL of water. Heat gently to dissolve. Dilute to 500mL with water.
6.3. *Ammonium thiocyanate indicator (1M)*-Dissolve 7.6g of ammonium thiocyanate in 90mL of water. Dilute to 100mL with water.
6.4. *Ba carrier* (5 mg/mL)- Dissolve 4.37g of $BaCl_2{\cdot}2H_2O$ in 250mL of water. Dilute to 500mL with water.
6.5. *Calcium nitrate (1.25M)*- Dissolve 102g of $Ca(NO_3)_2$ in 400mL of water. Dilute to 500mL with water.
6.6. *Hydrochloric acid (1M)* - Add 83mL of concentrated HCl to 800mL of water. Dilute to 1L with water
6.7. *Hydrochloric acid (4M)*- Add 333mL of concentrated HCl to 500mL of water. Dilute to 1L with water.
6.8. *Hydrochloric acid (4M)- hydrofluoric acid (0.1M)*- Add 333mL of concentrated HCl and 3.6mL of concentrated HF to 500mL of water. Dilute to 1L with water.
6.9. *Hydrochloric acid (5M)- oxalic acid (0.05M) solution*- Dissolve 6.3g oxalic acid dihydrate in 400mL of water. Add 417mL concentrated HCl. Dilute to 1L with water.
6.10. *Hydrochloric acid (9M)*- Add 750mL of concentrated HCl to 100mL of water. Dilute to 1L with water.
6.11. *Nitric acid (2M)- sodium nitrite (0.1M) solution*- Add 31mL of concentrated HNO_3 to 200mL of water, dissolve 1.72g of sodium nitrite in the solution. Dilute to 250mL with of water. **Prepare fresh daily.**
6.12. *Nitric acid solution (3M)*- Add 188mL of concentrated HNO_3 to 700mL of water. Dilute to 1L with water.
6.13. *Nitric acid (8M)* - Add 500mL of concentrated HNO_3 to 45 mL of water. Dilute to 1L with water.
6.14. *Nitric acid (3M) - Aluminum nitrate (1.0M) solution*- Dissolve 375g of $Al(NO_3)_3{\cdot}9H_2O$ in 500mL of water. Add 188mL of concentrated HNO_3. Dilute to 1L with of deionized water.
6.15. *Ferric Nitrate Solution (5 mg/mL Fe) in 0.1M HNO_3* – To a 500mL volumetric flask, add 18g $Fe(NO_3)_3{\cdot}9H_2O$, 400mL water and 3.1mL concentrated HNO_3. Swirl to dissolve. Dilute to 500mL with water.

REFERENCES

(1) Horwitz, E.P., et al. "Separation and Preconcentration of Actinides from Acidic Media by Extraction Chromatography," *Analytica Chimica Acta*, 281, 361-372, (1993).

(2) Horwitz, E.P., et al. "Separation and Preconcentration of Uranium from Acidic Media by Extraction Chromatography," *Analytica Chimica Acta*, 266, 25-37, (1992).

(3) Maxwell, S.L., et al. "Rapid Analysis of Urine and Water Samples," *Journal of Radioanalytical and Nuclear Chemistry*, 275(3), 447-502 (2008).

(4) ASTM Method D3648-14, "Standard Practices for the Measurement of Radioactivity."

(5) ASTM Method D7282-06, "Standard Practice for Set-up, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements."

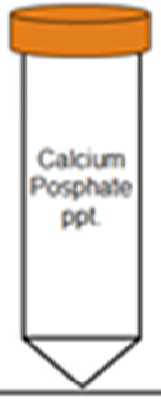
ASTM International, Standards for Radiochemistry, http://www.astm.org/

National Analytical Management Program (NAMP), Webinar Series on Actinide chemistry, http://www.wipp.energy.gov/namp/en_content-30-trainingedu.html

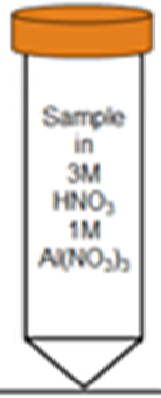
National Academy of Sciences, Monograph Series on Radiochemistry, http://www.lanl.gov/library/find/ebooks/radiochem.php

1) Aliquot up to 1200mL of urine into glass beaker.
2) Add yield tracers.
3) Add 2-3 drops 1-octanol, 1mL of 1.25M $Ca(NO_3)_2$, and 1mL Ba carrier.
4) Add 25mL of conc. HNO_3.
5) Cover with watch glass and heat at medium setting for at least 30-60 min.
6) Turn down heat. Add 2.5mL of 3.2M $(NH_4)_2HPO_4$.
7) While stirring sample, slowly add conc. NH_4OH until reaching pH 9.
8) After 30 minutes, turn off heat. Allow sample to cool and precipitate to settle or centrifuge.
9) Decant supernate and discard as waste.
10) Transfer precipitate to centrifuge tube with DI water.
11) Centrifuge ~10minutes at 2000rpm. Decant supernate.
12) Add 10mL DI water to precipitate. Mix well. Centrifuge. Decant supernate.
13) Rinse precipitation beaker with 5mL conc. HNO_3. Tranfer to precipitate in centrifuge tube. Dissolve precipitate and transfer to 100mL beaker.
14) Rinse centrifuge tube with 2x 5mL conc. HNO_3. Transfer to same 100mL beaker.
15) Digest precipitate with HNO_3/H_2O_2 until white residue obained.
16) Dissolve reside in 20mL 3M HNO_3-1M $Al(NO_3)_3$.
17) Add 1mL 1.5M Sulfamic Acid, 0.5 mL Fe, and 1mL 1M Ascorbic Acid.

Urine Sample in glass beaker. HNO_3 digest.



Digest residue in glass beaker with conc. HNO_3/H_2O_2



18) Precondition UTEVA-TRU with 5mL 3M HNO_3.
19) Load sample onto UTEVA-TRU. Allow liquid to drain. UTEVA retaines U. TRU retains Am and Pu.
20) Rinse sample tube with 5mL 3M HNO_3. Add rinse to UTEVA-TRU. Allow liquid to drain.
21) Rinse UTEVA-TRU with 5mL 3M HNO_3. Allow liquid to drain.
22) Separate UTEVA and TRU cartridges.

(18) (19) (20) (21) → Waste

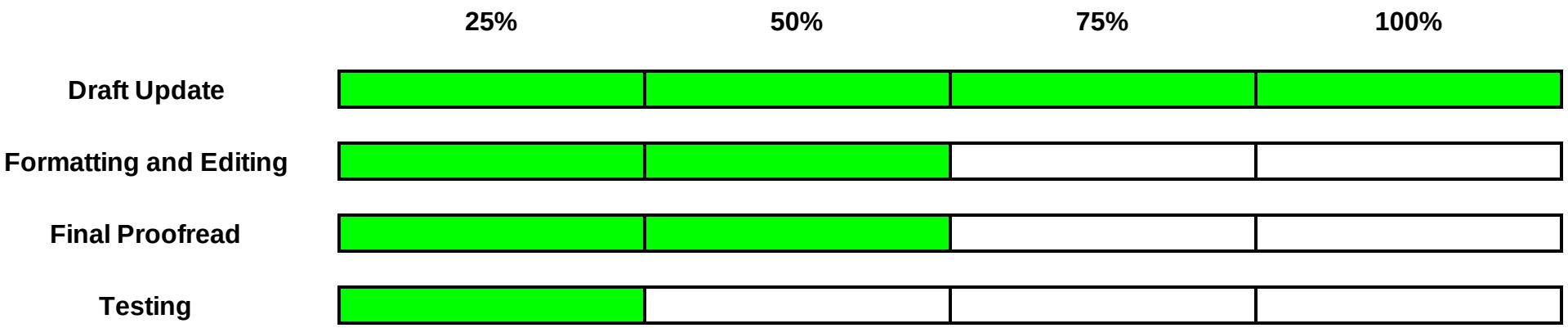
23) Rinse UTEVA cartridge with 15mL 8M HNO_3.
24) Rinse UTEVA cartridge with 5mL 9M HCl.
25) Rinse UTEVA cartridge with 20mL 5M HCl-0.05M oxalic acid. Discard to Waste.
26) Place clean centrifuge tube below each cartridge. Strip Uranium with 15mL 1M HCl.

(23) (24) (25) → Waste
(26) → U sample to source preparation

27) Rinse TRU with 5mL 2M HNO_3-0.1M $NaNO_2$.
28) Place clean centrifuge tube below each cartridge. Strip Am with 15mL 4M HCl.
29) Place a clean centrifuge tube below each cartridge. Rinse TRU with 25mL 4M HCl-0.1M HF.
30) Place a clean centrifgue tube below each cartridge. Strip Pu with 10mL 0.1M ammonium bioxalate.

(27) (29) → Waste
(28) → Am sample to source preparation
(30) → Pu sample to source preparation

Update Progress



Application Notes

- Significant changes to the chemistry

- Application to new matrix or sample size

- New analyte or combination of analytes

- Make vast library of applications of Eichrom Products more accessible.

- Two pages highlight:
  - Summary of method
  - Reagent/Equipment lists
  - Flowsheet(s)
  - Testing data
  - Reference to literature citation

eichrom

AN-1404

Rapid Determination of Sr in 1-2 Liter Seawater Samples

Summary of Method Strontium is separated and concentrated from 1-2L samples of seawater with a calcium phosphate precipitation, enhanced with 200mg of iron and 1mg of yttrium. Strontium is separated from matrix impurities and potentially interfering radionuclides in the sample using two stacked 2mL cartridges of Eichrom Sr Resin. Radiostrontium is measured on a low background gas flow proportional counter. Chemical yield of strontium is determined by gravimetric recovery of native stable strontium in the seawater. Average chemical recovery of strontium is 89 ± 5% for 1L samples and 82 ± 4% for 2L samples. Measured values of ^{90}Sr agreed to within 1% and 4% of reference values, for 1L and 2L, respectively, with two hour count times. The minimum detectable activity for ^{90}Sr for 2L samples with a two hour count time is 9.1Bq/L. A single operator can prepare batches of 12-24 samples for measurement of radiostrontium in less than 8 hours.

Reagents

Sr Resin, 2mL Cartridges (Eichrom SR-R50-S)
Nitric Acid (70%)
Ammonium Hydroxide (57%)
Deionized Water
Iron Carrier (50mg/mL Fe, as ferric nitrate)
Yttrium Carrier (1mg/mL)
3.2M $(NH_4)_2HPO_4$
Aluminum Nitrate, Nonahydrate
^{90}Sr standard
Oxalic acid
Boric acid

Equipment

Vacuum Box (Eichrom AC-24-BOX or AC-12-BOX)
Inner Support Tubes-PE (Eichrom AC-1000-TUBE-PE)
Yellow Outer Tips (Eichrom AC-1000-OT)
50mL Centrifuge Tubes
250-500mL Centrifuge Tubes
Centrifuge
Cupped Stainless Steel Planchets (~5mL volume)
Gas Flow Proportional Counter
Hot Plate
Analytical Balance
600mL Glass Beakers
Vacuum Pump

Figure 1. Sample Preparation

1-2L Sample of Seawater. Add 200mg Fe(III), 1mg Y(III), and 25mL 3.2M $(NH_4)_2HPO_4$.

↓ Mix Well.

↓ Adjust to pH 10 with ~30mL of 57% NH_4OH while stirring.

↓ Centrifuge for 10 minutes in 250 or 500mL tubes at 3000rpm, decanting supernate to waste, until entire sample has been processed.

↓ Wash precipitate with 50mL of deionized water

↓ Centrifuge for 10 minutes at 3000rpm. Decant supernate to waste

↓ Dissolve precipitate in 25mL 70% HNO_3. Transfer to 600mL glass beaker.

↓ Rinse centrifuge tube with 8mL 2M $Al(NO_3)_3$ and 5mL 3M HNO_3. Transfer tube rinse to Sr samples in 600mL glass beaker.

↓ Evaporate samples to ~25mL on hot plate. Transfer samples to 50mL centrifuge tubes.

↓ Rinse beakers with 2 x 5mL 1M HNO_3. Transfer beaker rinses to samples in 50mL centrifuge tubes.

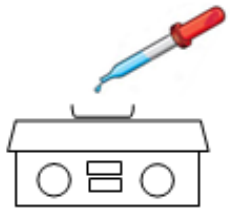
↓ Load Solution to Sr Resin Separation

Figure 2. Strontium Resin Separation (Optional ^{90}Y Ingrowth)

(1) Precondition Sr Resin with 10mL 8M HNO_3.
(2) Load ^{90}Sr sample at 1-2mL/min.
(3) Rinse sample tube with 5mL 8M HNO_3.
(4) Add tube rinse to Sr Resin. Elute at 1-2mL/min.
(5) Rinse Sr Resin sequentially with:
- 15mL 8M HNO_3
- 10mL 3M HNO_3 - 0.05 oxalic acid
- 10mL 8M HNO_3

(6) Dispose of (1) to (5) as waste.
(7) Strip Sr with 20mL 0.05M HNO_3 at 1mL/min.

(8) Evaporate samples to dryness on tared cupped stainless steel planchets.
(9) Rinse Sr sample vials with 2mL 0.05M HNO_3. Transfer vial rinse to planchets. Evaporate to dryness.



(10) Weigh planchets on an analytical balance to determine gravimetric yield of stable $Sr(NO_3)_2$.
(11) Measure radiostrontium in samples on low background gas flow proportional counter.

(Options for $^{89/90}$Sr Descrimination)

When necessary to obtain ^{89}Sr and ^{90}Sr data:

(a) Sr fraction from step (7) can be transfered to a liquid scintillation vial. ^{89}Sr can be measured by Cerenkov counting (without LSC cocktail).

(b) Sr fraction from step (10) can be dissolved in 10mL 8M HNO_3 after >7 days of ^{90}Y ingrowth. $^{89/90}$Sr can be removed on Sr Resin. ^{90}Y will elute in Sr Resin load and can be counted by liquid scintillation or gas flow proportional counting.

Performance of ^{90}Sr Method for 1L and 2L Seawater Samples

Sample Replicates	Sample Volume, L	^{90}Sr, Reference Value (mBq/L)	^{90}Sr, Measured Value (mBq/L)	% Bias	Sr carrier % Recovery
11	1	148	150 ± 11	1.2	89 ± 5
4	2	148	154 ± 5	4.2	82 ± 4

2 hour count times
MDA = 9.1 mBq/L for 2L sample

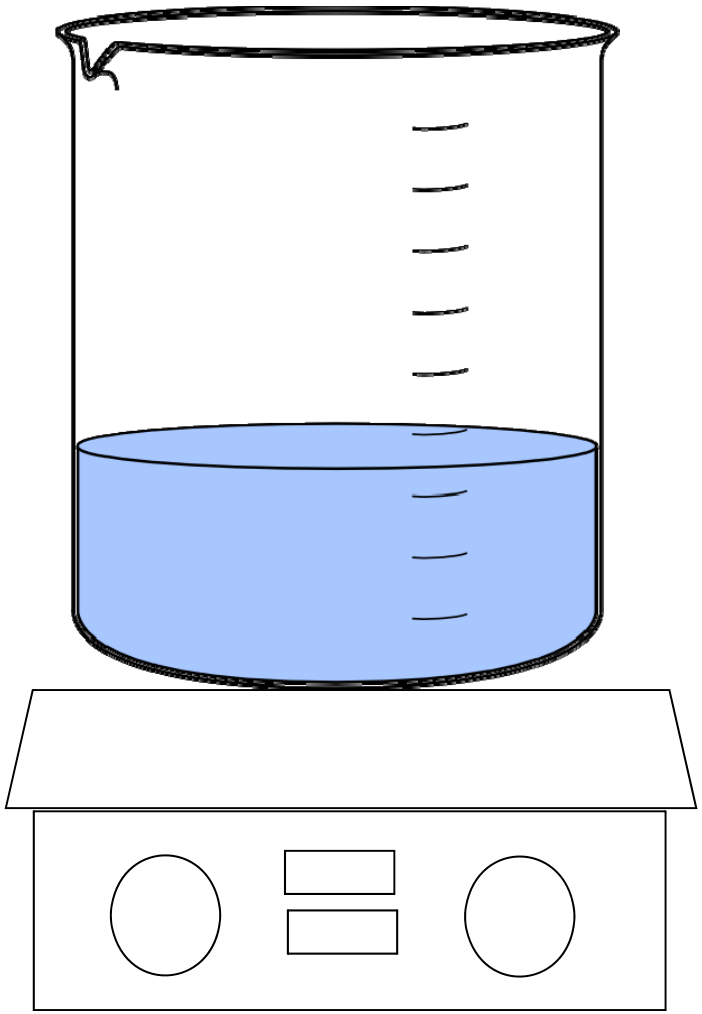
References

1) Sherod L. Maxwell, Brian K. Culligan, Robin C. Utsey, "Rapid determination of radiostrontium in seawater samples," *J. Radioanal. Nucl. Chem., 298(2), 867-875* (2013).

eichrom 1955 University Lane Lisle, IL 60532, USA +1 (630)963-0320 www.eichrom.com

Number	Title	Analytes	Matrix
AN-1401	Rapid Determination of ^{226}Ra in Emergency Urine and Water	^{226}Ra	Emergency Urine/Water
AN-1402	Rapid Determination of Sr in Emergency Milk Samples	Sr	Emergency Milk
AN-1403	Rapid Determination of Sr in 50g Soil Samples	Sr	Soil (50g)
AN-1404	Rapid Determination of Sr in 1-2 Liter Seawater Samples	Sr	Seawater (1-2L)
AN-1405	Rapid Determination of Sr in Vegetation Samples	Sr	Vegetation
AN-1406	Rapid Determination of Actinides in Vegetation Samples	U, Pu, Am, Cm	Vegetation
AN-1407	Rapid Determination of Sr in Animal Tissue Samples	Sr	Animal Tissue
AN-1408	Rapid Determination of Actinides in Animal Tissue Samples	U, Pu, Am, Cm	Animal Tissue
AN-1409	Rapid Determination of Sr in Building Materials	Sr	Building Materials
AN-1410	Rapid Determination of Sr in Emergency Urine Samples	Sr	Emergency Urine
AN-1411	Rapid Determination of Sr in Emergency Water Samples	Sr	Emergency Water
AN-1412	Rapid Determination of Actinides in Emergency Urine Samples	U, Pu, Am, Cm	Emergency Urine
AN-1413	Rapid Determination of Actinides in Emergency Water Samples	U, Pu, Am, Cm	Emergency Water
AN-1414	Rapid Determination of ^{90}Sr in Up to 40 Liter Seawater Samples	^{90}Sr	Seawater (40L)
AN-1415	Rapid Determination of ^{210}Po in Water Samples	^{210}Po	Water
AN-1416	Rapid Determination of Actinides and ^{210}Po in Water	^{210}Po, U, Pu, Am, Cm	Water
AN-1417	Rapid Determination of $^{226/228}$Ra in Water Samples	$^{226/228}$Ra	Water
AN-1418	Rapid Determination of ^{226}Ra in Water Samples	^{226}Ra	Water
AN-1419	Rapid Determination of ^{226}Ra in Concrete and Brick	^{226}Ra	Concrete/Brick
AN-1420	Rapid Determination of 226Ra in Glass Fiber Air Filters	^{226}Ra	Glass Fiber Air Filters
AN-1421	Rapid Determination of 226Ra in 1g Soil Samples	^{226}Ra	Soil
AN-1422	Rapid Determination of 226Ra in 5g Vegetation Samples	^{226}Ra	Vegetation
AN-1423	Rapid Determination of Pu, Np, and U in 1-8L Seawater Samples	Np, Pu, U	Seawater (1-8L)
AN-1424	Rapid Determiantion of Pu, Am and Cm in 80L Seawater Samples	Pu, Am, Cm	Seawater (80L)
AN-1425	Rapid Determination of Actinides in 10g Emergency Food Samples	U, Pu, Np, Am, Cm	Emergency Food (10g)
AN-1426	Rapid Determination of Actinides in 100g Emergency Food Samples	U, Pu, Np, Am, Cm	Emergency Food (100g)
Coming Soon			
AN-1427	Rapid Method for Plutonium in Rice	Pu	Rice
AN-1428	Rapid Method for Actinides in fecal samples	Am, Pu, U	Fecal Samples
AN-1429	Rapid Method for Actinides in Asphalt samples	Am, Pu, U	Asphalt
AN-1430	Rapid Method for Actinides in Emergency Soil Samples	Am, Cm, Pu, U	Emergency Soil
AN-1431	Rapid Method for Determination of Pu, Am, Cm in large soil samples	Pu, Am, Cm	100-200g Soil
AN-1432	Rapid Method for Actinides in Emergency Concrete and Brick Samples	Am, Cm, Pu, Np, U	Concrete and Brick
AN-1433	Rapid Method for actinides in emergency air filter samples	Pu, Am, U	Air Filters
AN-1434	Rapid Method for strontium in emergency air filter samples	Sr	Air Filters
AN-1435	Rapid Method for Np and Pu in large soil samples	Np, Pu	20-50g Soil
AN-1436	Rapid Method for Np and Pu in large soil samples by ICP-MS	Np, Pu	20-75g Soil
AN-1437	Rapid Method for Actinides in Urine by ICP-MS and alpha spectrometry	Am, Cu, Pu, Np, U	urine
AN-1438	Rapid Method for Np and Pu in water by ICP-MS and alpha specrometry	Np, Pu	water

Sample Preparation



1) Acidify with HNO_3 (pH 1-2).

2) Add Tracers.

3) Heat. Mix.

4) Remove from heat.

5) Add Ca/$(NH_4)_2HPO_3$.

6) Mix. Add NH_4OH to pH 8-9

7) Additional heating.

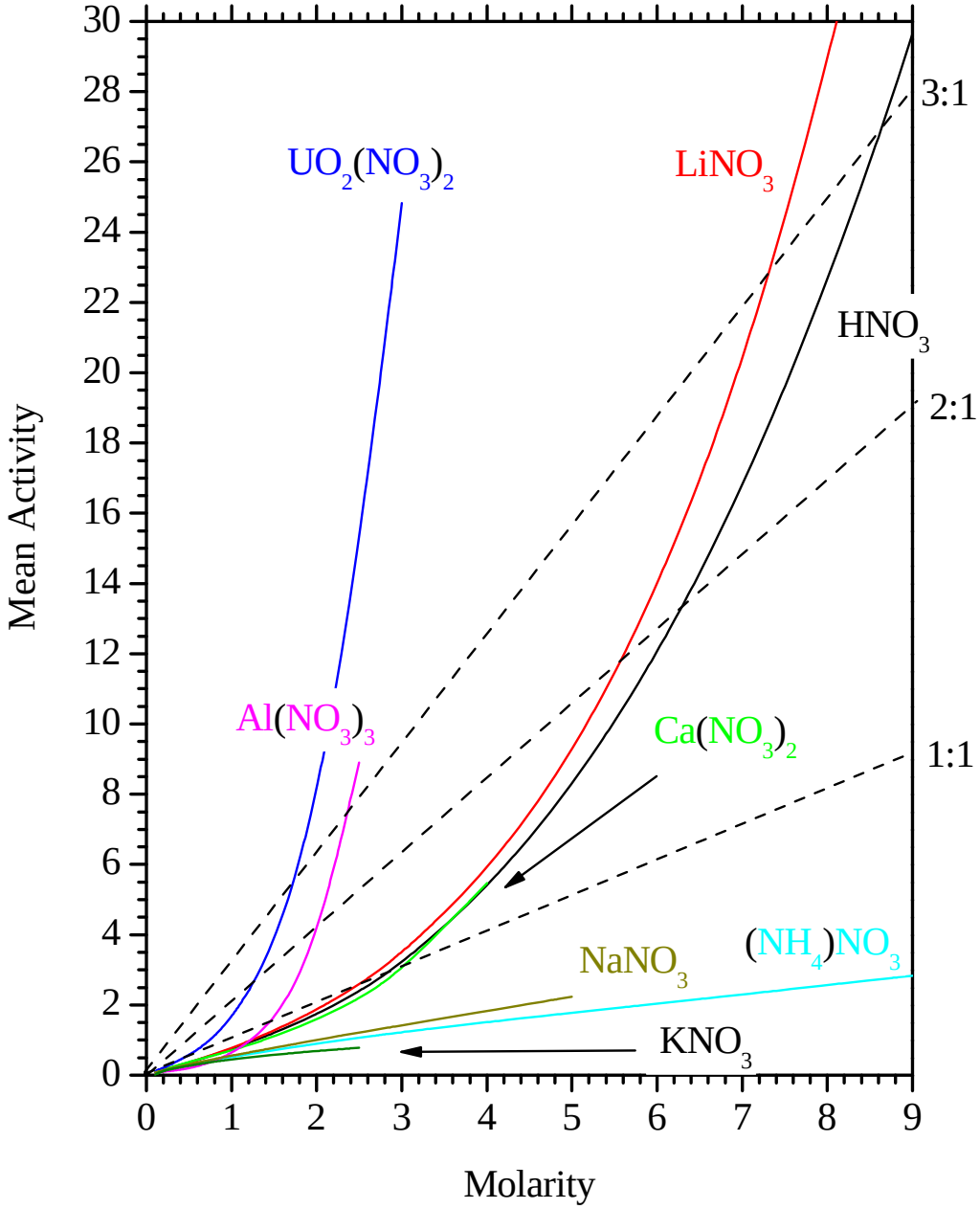
8) Cool. Settle. Centrifuge.

9) Wash ppt. w/ dilute NH_4OH.

$Al(NO_3)_3$-HNO_3

1) HNO_3 protonates PO_4^{3-}, dissolving calcium phosphate ppt.

2) $Al(NO_3)_3$ – HNO_3 provide NO_3^- to promote extraction of nitrato-complexes of actindes/Sr.


Mean Activity
Molarity
0
2
4
6
8
10
12
14
16
18
20
22
24
26
28
30
0
1
2
3
4
5
6
7
8
9
UO2(NO3)2
LiNO3
HNO3
3:1
2:1
1:1
Al(NO3)3
Ca(NO3)2
NaNO3
(NH4)NO3
KNO3

$Al(NO_3)_3$-HNO_3

1) HNO_3 protonates PO_4^{3-}, dissolving calcium phosphate ppt.

2) $Al(NO_3)_3$ – HNO_3 provide NO_3^- to promote extraction of nitrato-complexes of actindes/Sr.

3) Al^{3+} complexes phosphate, minimizing impact on Th^{4+}.

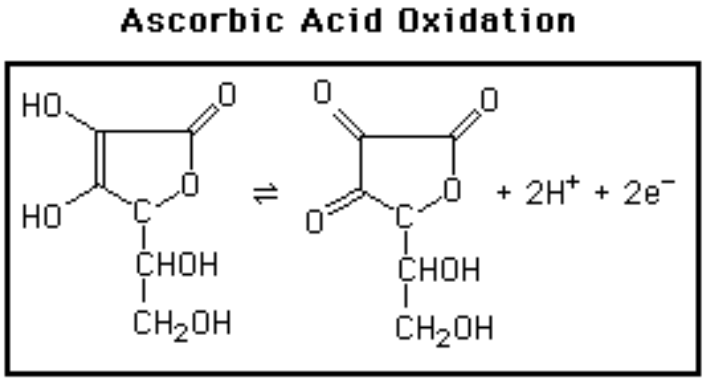
Valence Adjustment

1) Cool to room temperature. Warm solutions can increase unwanted reactions.

2) Add ferric iron and sulfamic acid. Mix.

$$HNO_2 + H_3NSO_3 \rightarrow H_2SO_4 + N_2 + H_2O$$
$$HNO_3 + H_3NSO_3 \rightarrow H_2SO_4 + N_2O + H_2O$$

3) Add ascorbic acid to reduce Fe(II), Pu(III), Np(IV). Uranium remains U(VI). Am/Cm(III). Sr(II).



4) Add $NaNO_2$ to oxidize Pu(III) to Pu(IV).

I.L. Jenkins, "Factors governing the choice of 237Np/238Pu Separation Process, *Actinides Reviews*, 1, 187-211 (1969)

Column/Cartridge Separations

1) Precondition w/ 2-3 bed volumes of 3M HNO_3.

2) Load/Rinse at 2mL/min.

3) OK for cartridges to run dry.

4) New/clean reservoirs and vacuum box tips.

5) Strip analytes at 1mL/min.

Alpha Spectrometry Source Preparation

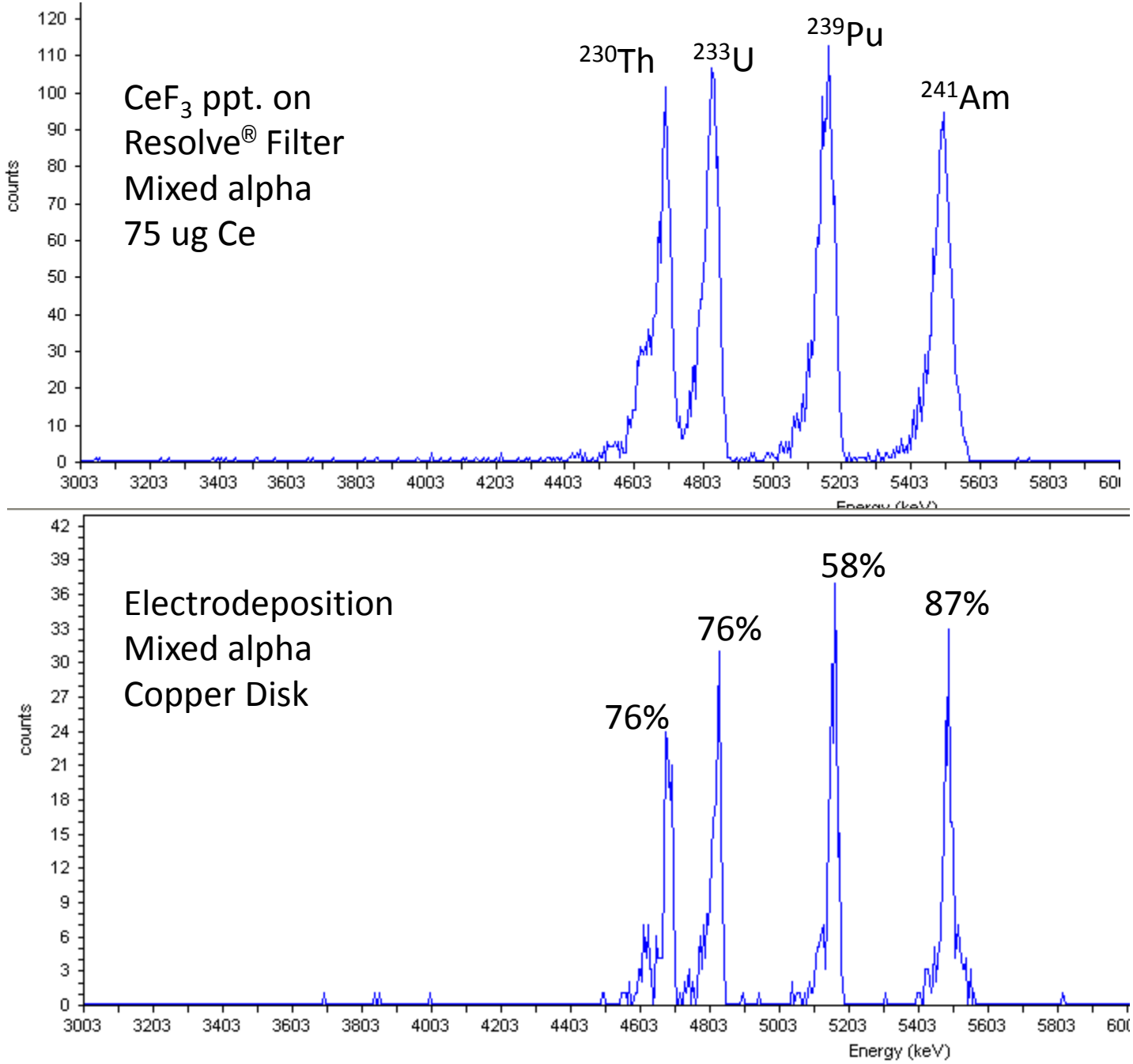
Microprecipitation	Electrodeposition
Adequate resolution	Superior resolution
Rapid/Many Samples	Slower
Direct Prep. from Column Strip Solution	Requires evaporation to remove HNO_3/HCl.
Most Routine Analytical Sources	Calibration Sources Nuclides with difficult to resolve peaks

Need Geometry Match to Calibration Source

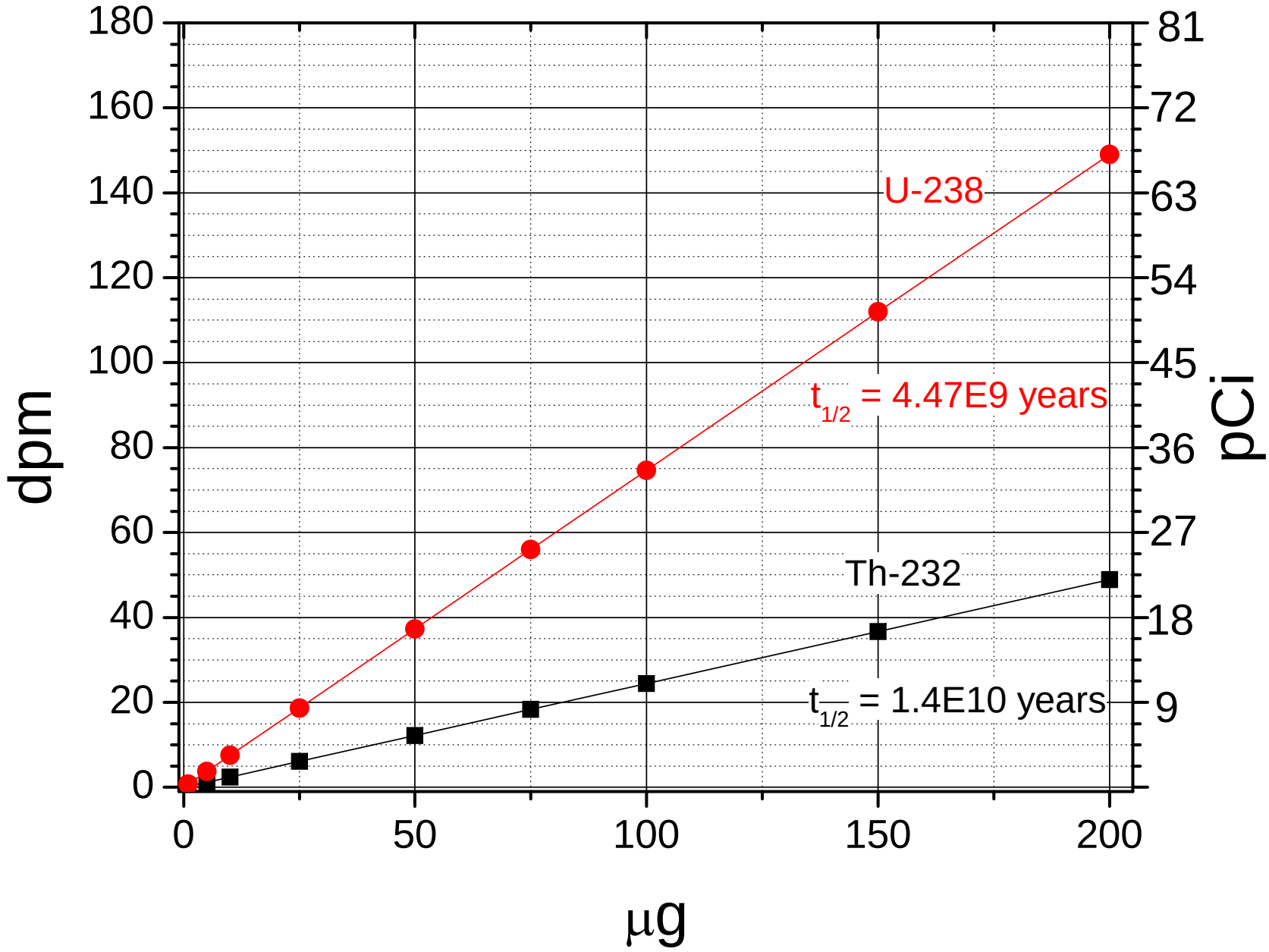
M. Schultz, NAMP Actinide Chemistry Webinar Series, "Source Preparation for alpha spectrometry," https://www.icln.org/default/assets//File/Source%20Prep%20Alpha%20Spec%20Final_1-21-13%20slide%20deck.pdf

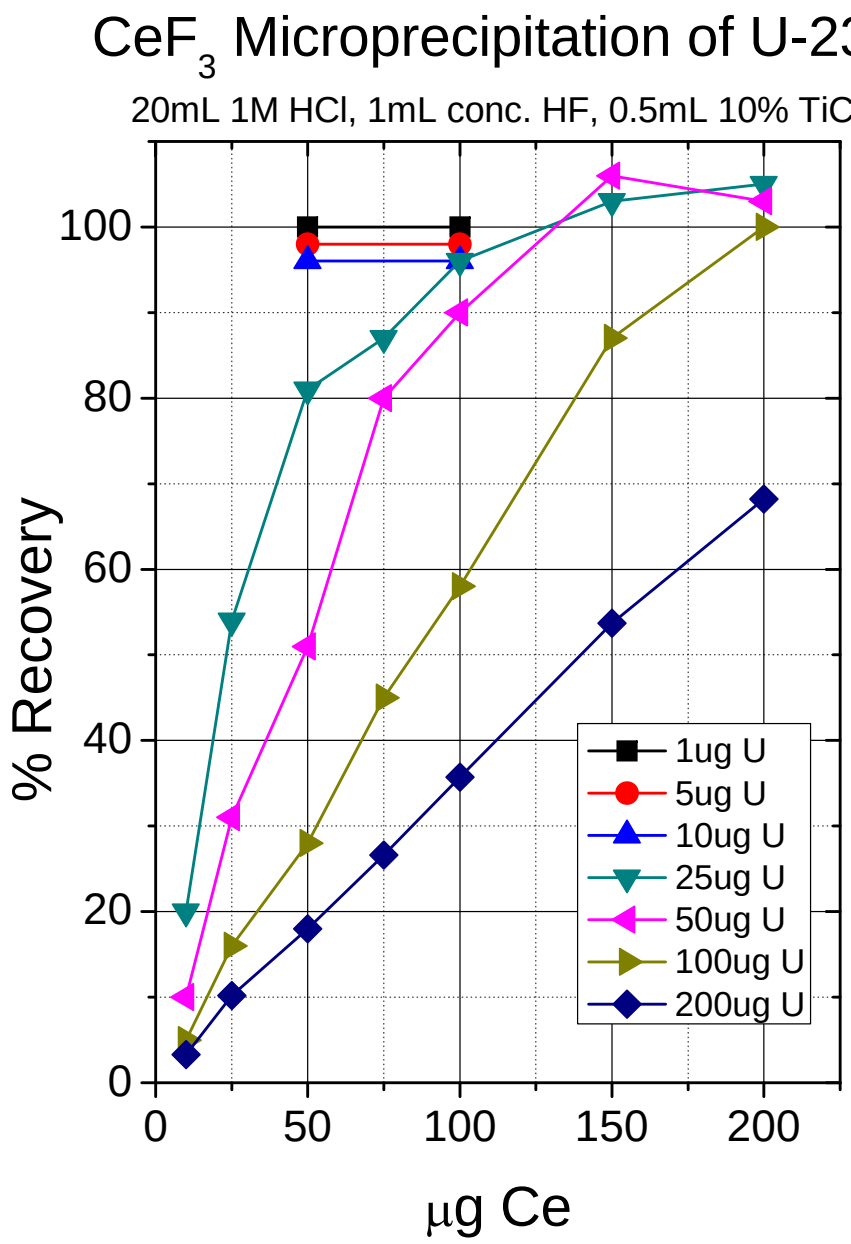
ASTM Method C1284-10 "Standard Practice for Electrodeposition of the Actinides for Alpha Spectrometry"

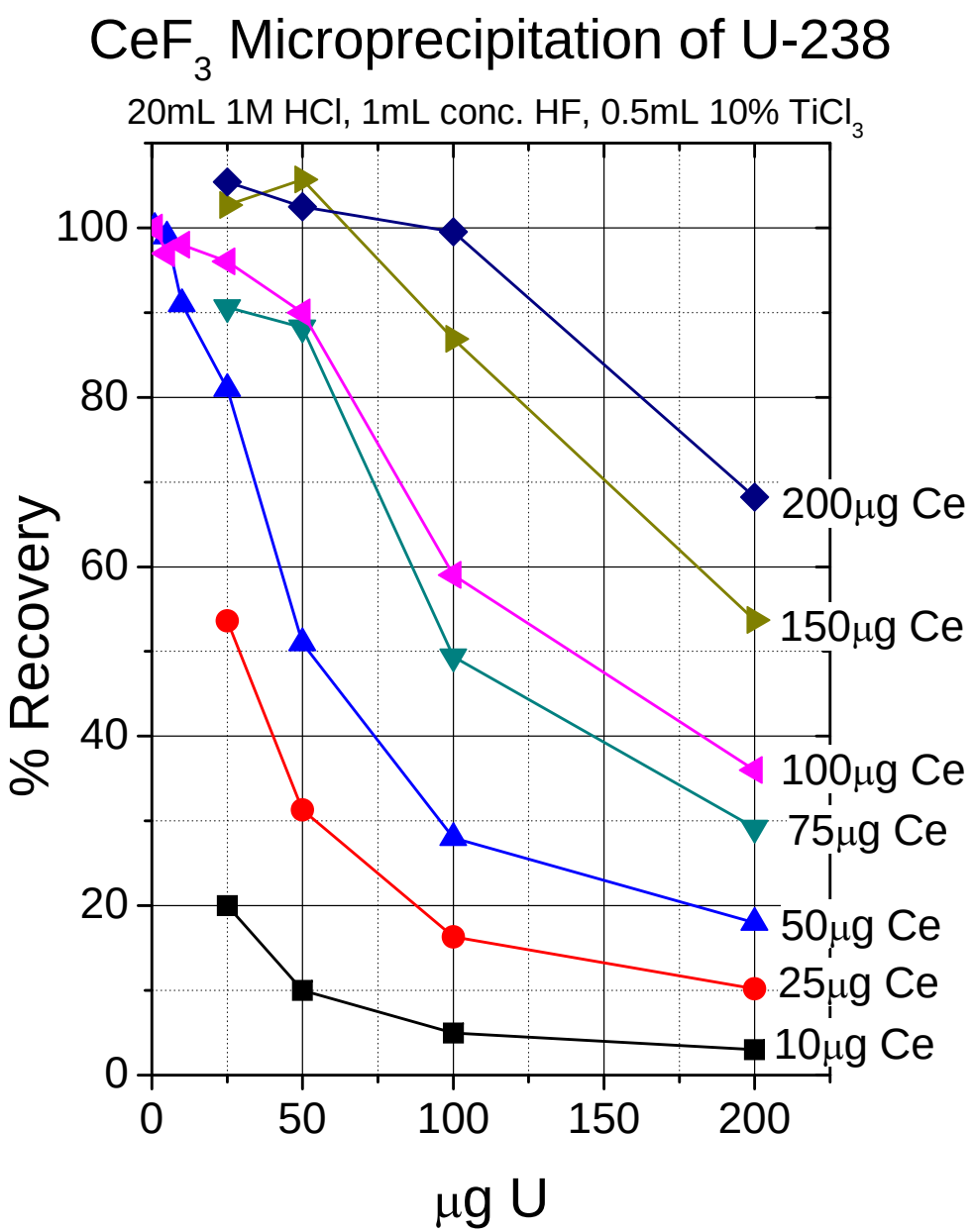
C. W. Sill, "Precipitation of actinides as fluorides or hydroxides for high-resolution alpha spectrometery," *Nuclear and Chemical Waste Management*, 7, 201-215 (1987).

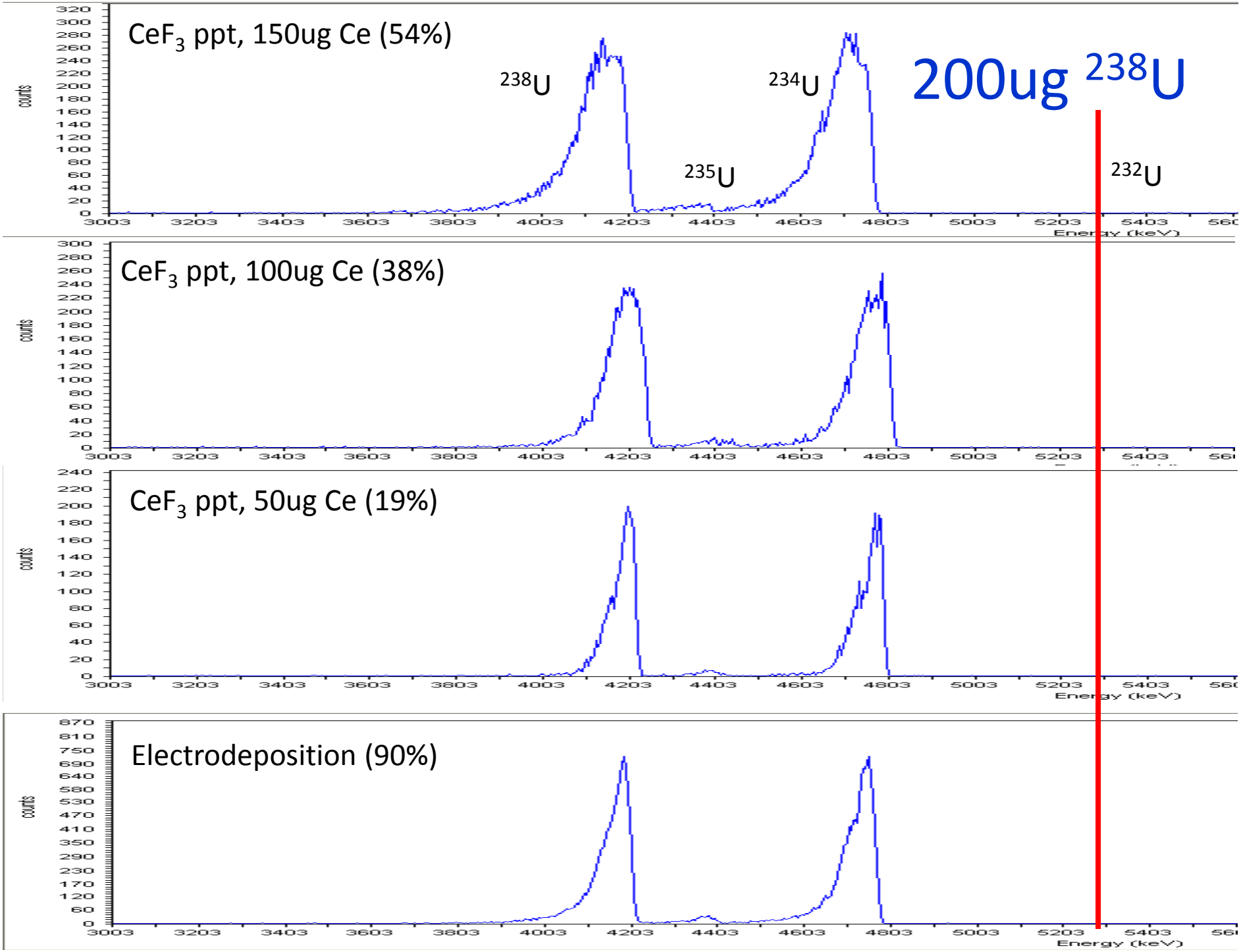


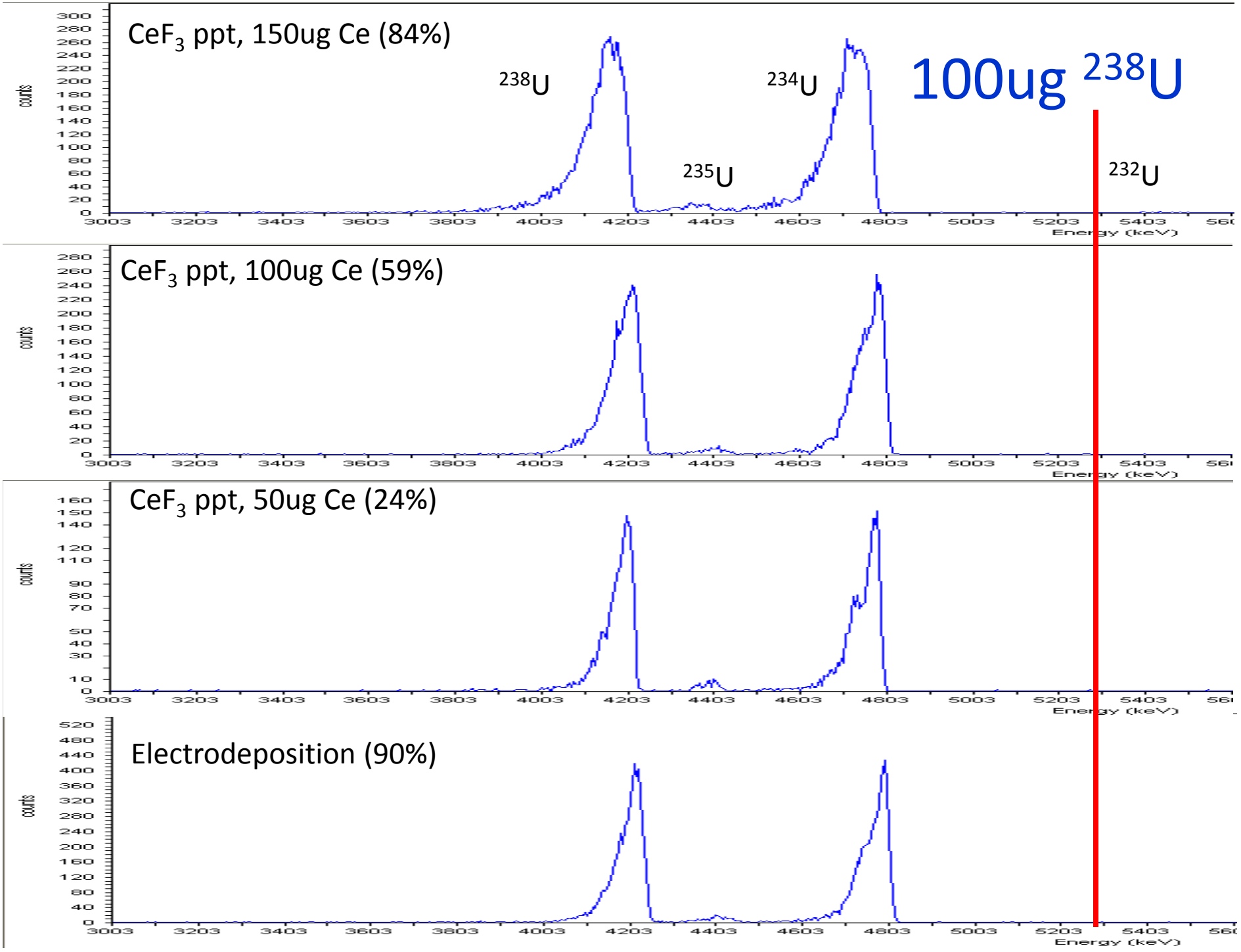
M. Schultz, NAMP Actinide Chemistry Webinar Series, "Source Preparation for alpha spectrometry," https://www.icln.org/default/assets//File/Source%20Prep%20Alpha%20Spec%20Final_1-21-13%20slide%20deck.pdf

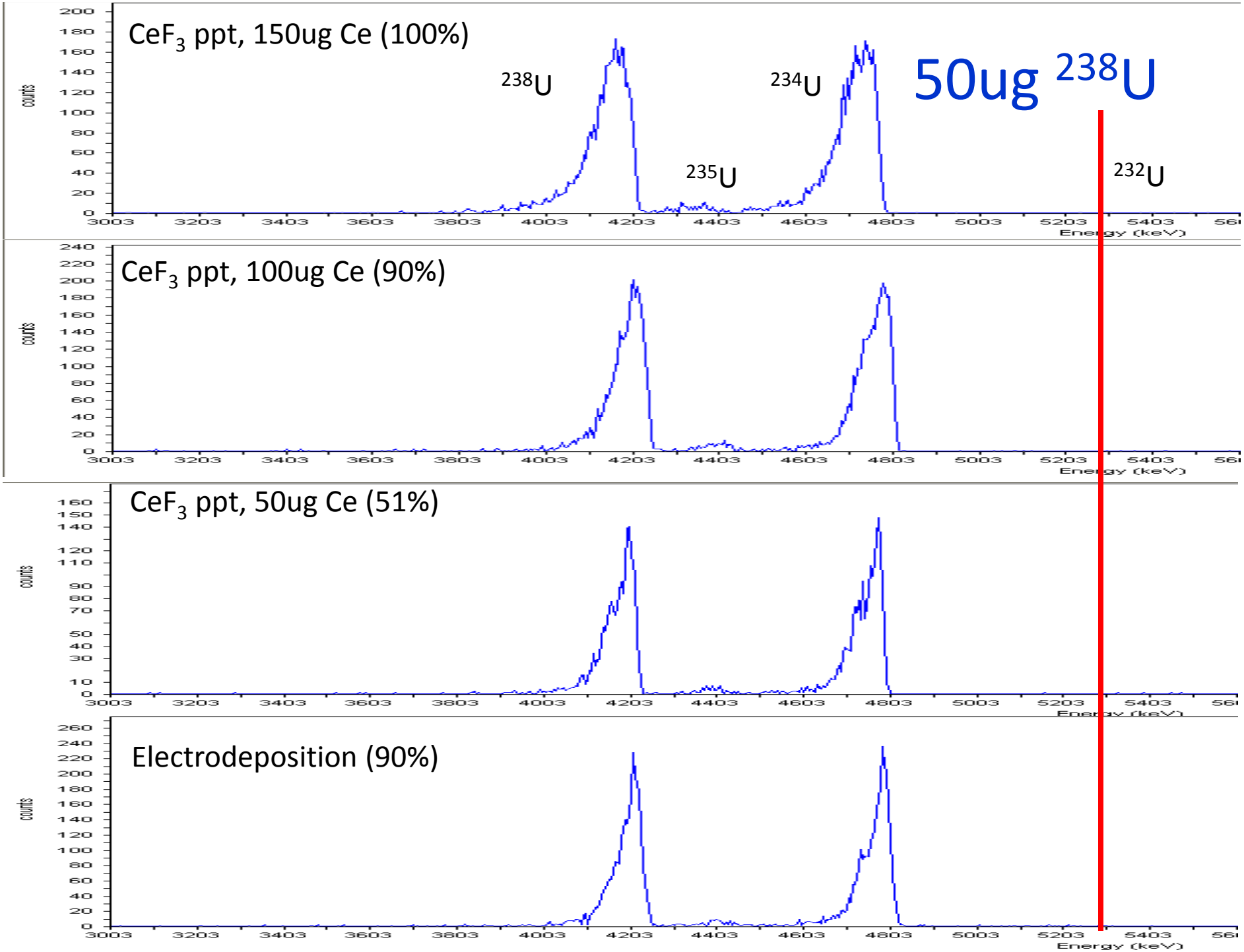

dpm
pCi
μg
U-238
$t_{1/2}$ = 4.47E9 years
Th-232
$t_{1/2}$ = 1.4E10 years
0
20
40
60
80
100
120
140
160
180
9
18
27
36
45
54
63
72
81
0
50
100
150
200

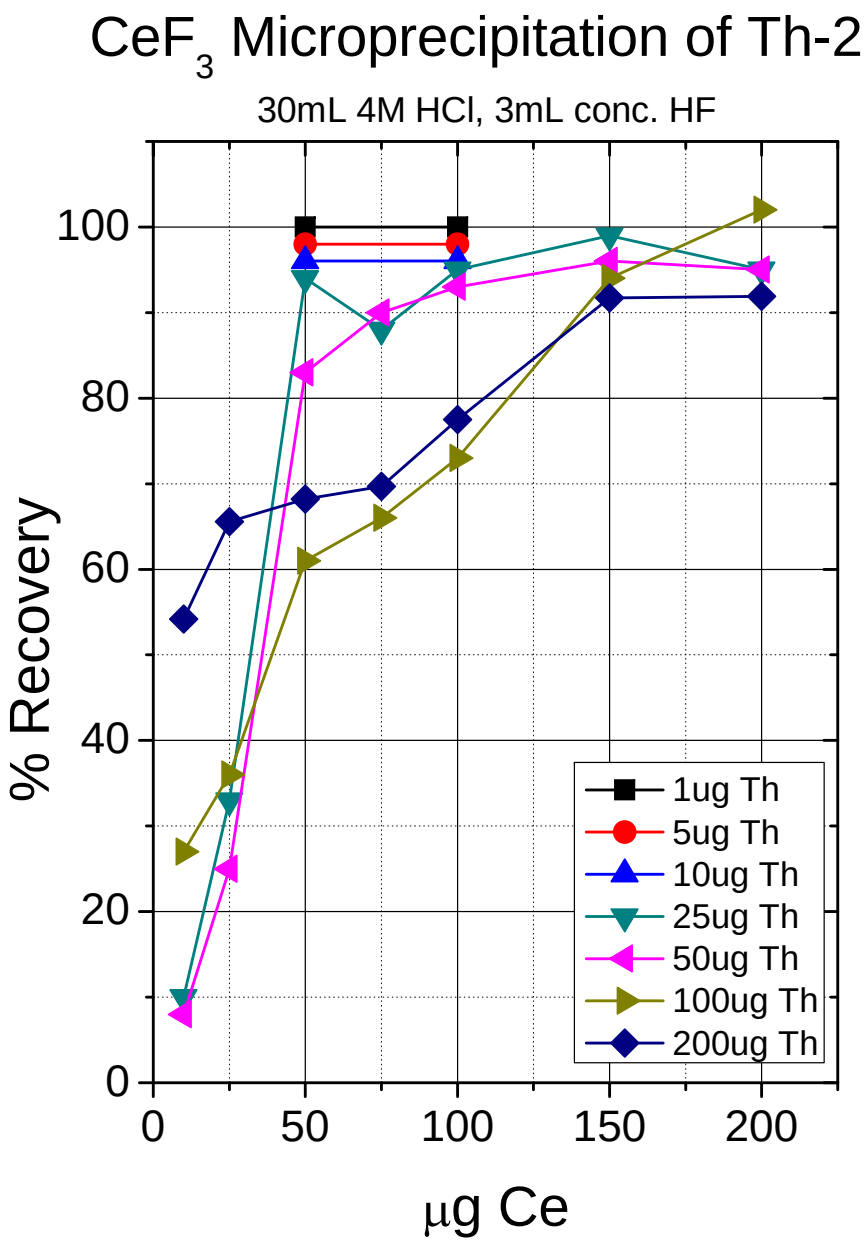

CeF3 Microprecipitation of U-238
20mL 1M HCl, 1mL conc. HF, 0.5mL 10% TiCl3
% Recovery
μg Ce
0
20
40
60
80
100
0
50
100
150
200
1ug U
5ug U
10ug U
25ug U
50ug U
100ug U
200ug U

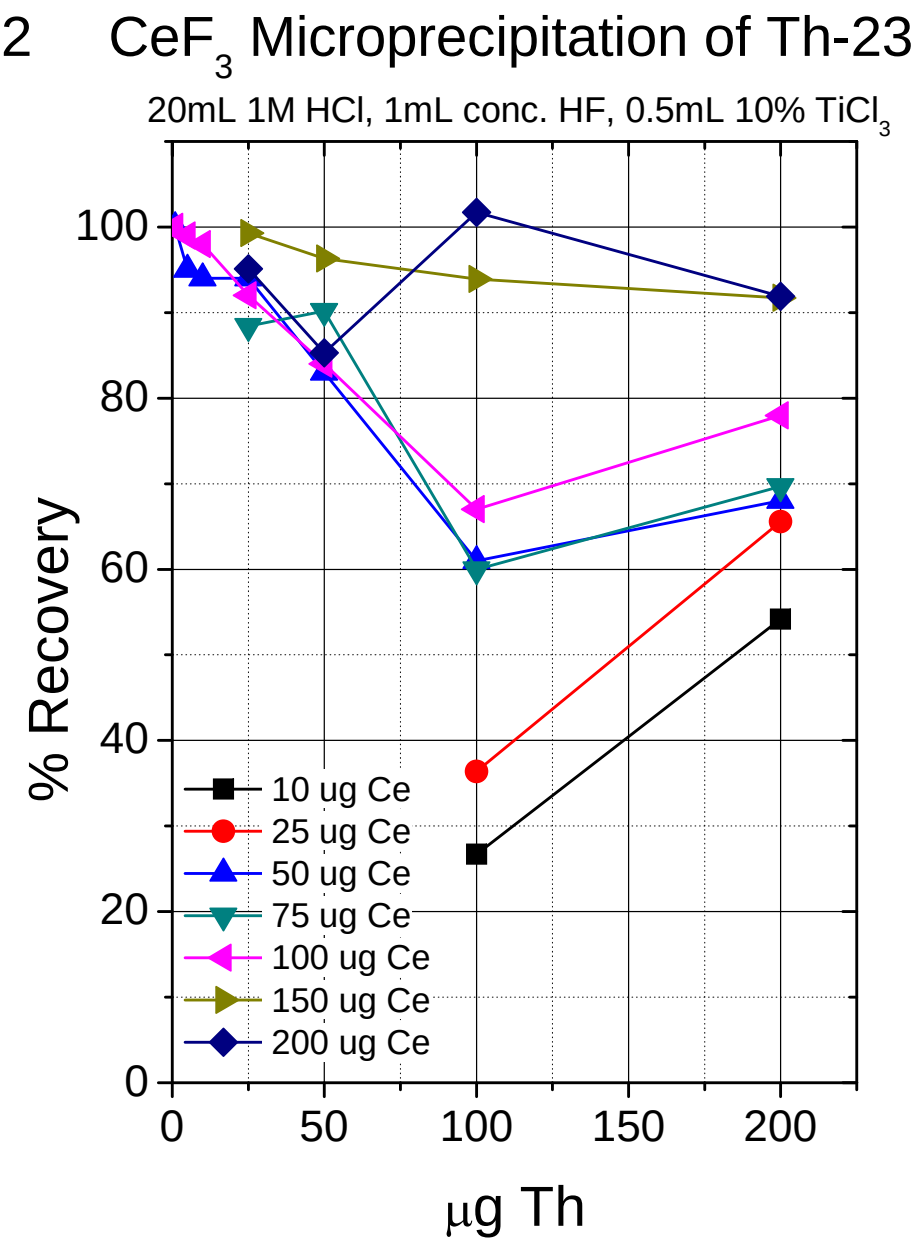

CeF3 Microprecipitation of U-238
20mL 1M HCl, 1mL conc. HF, 0.5mL 10% TiCl3
% Recovery
μg U
0
20
40
60
80
100
0
50
100
150
200
200μg Ce
150μg Ce
100μg Ce
75μg Ce
50μg Ce
25μg Ce
10μg Ce

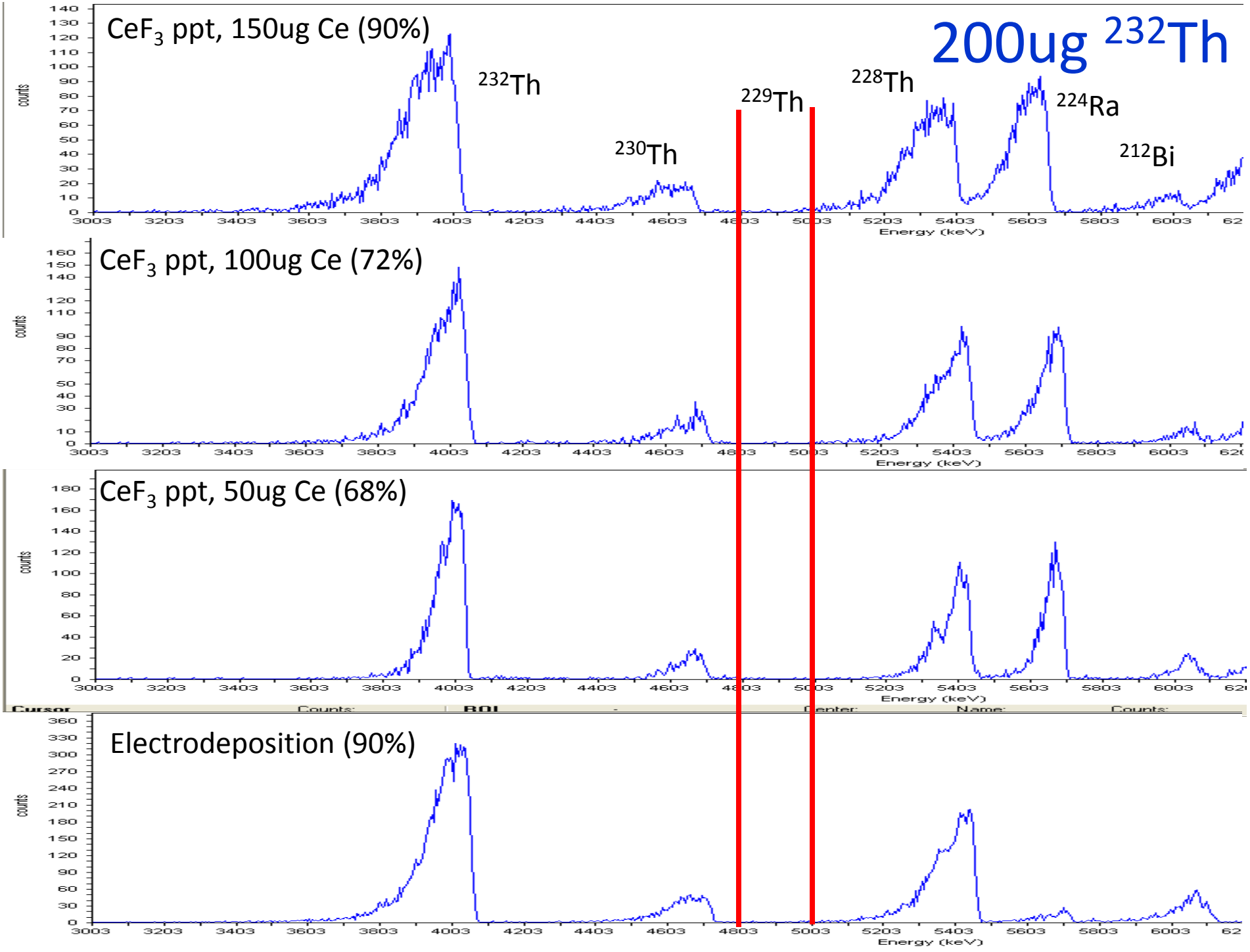

CeF3 ppt, 150ug Ce (54%)
238U
234U
235U
232U
200ug 238U
CeF3 ppt, 100ug Ce (38%)
CeF3 ppt, 50ug Ce (19%)
Electrodeposition (90%)
counts
Energy (keV)

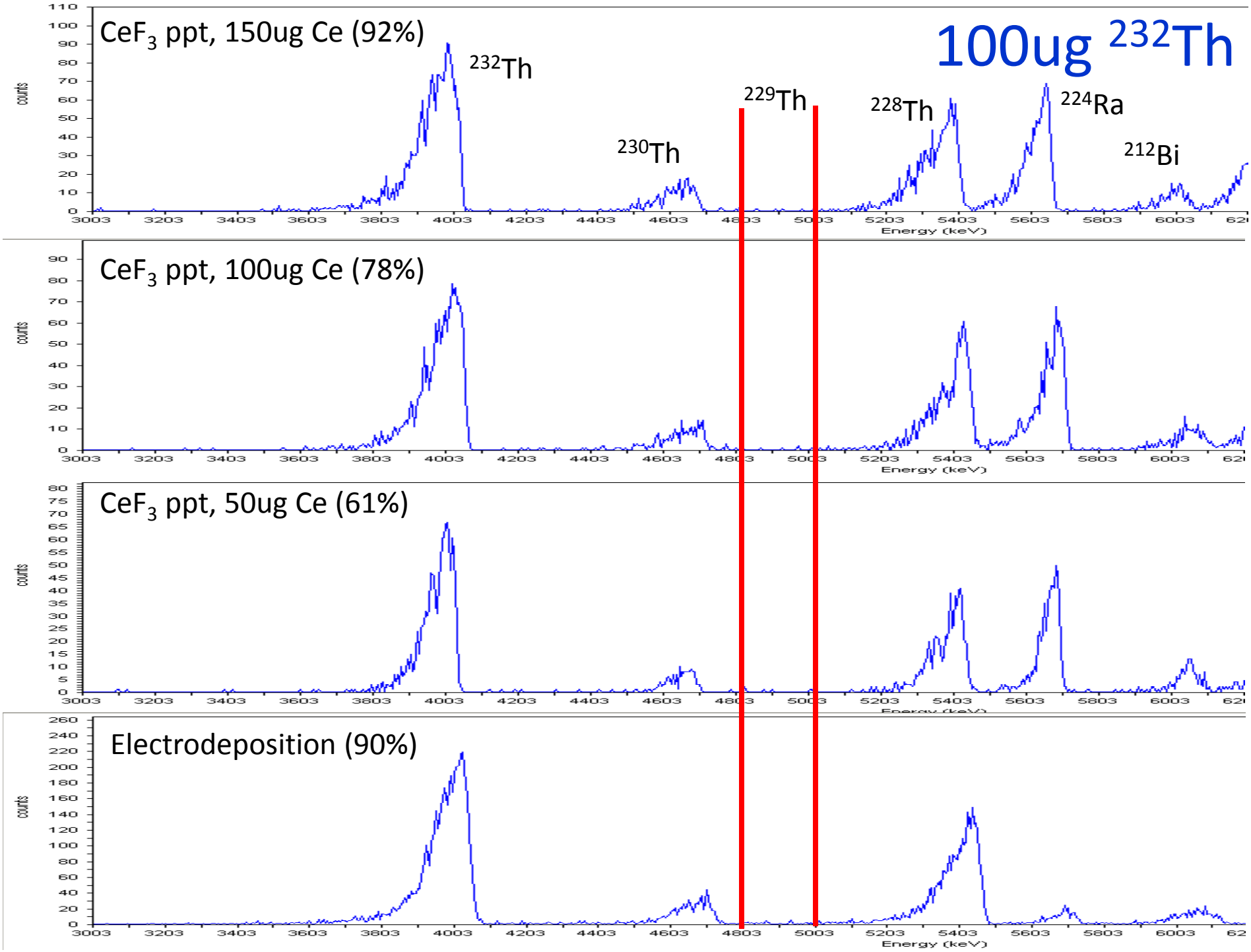

CeF3 ppt, 150ug Ce (84%)
238U
234U
235U
232U
100ug 238U
CeF3 ppt, 100ug Ce (59%)
CeF3 ppt, 50ug Ce (24%)
Electrodeposition (90%)
counts
Energy (keV)

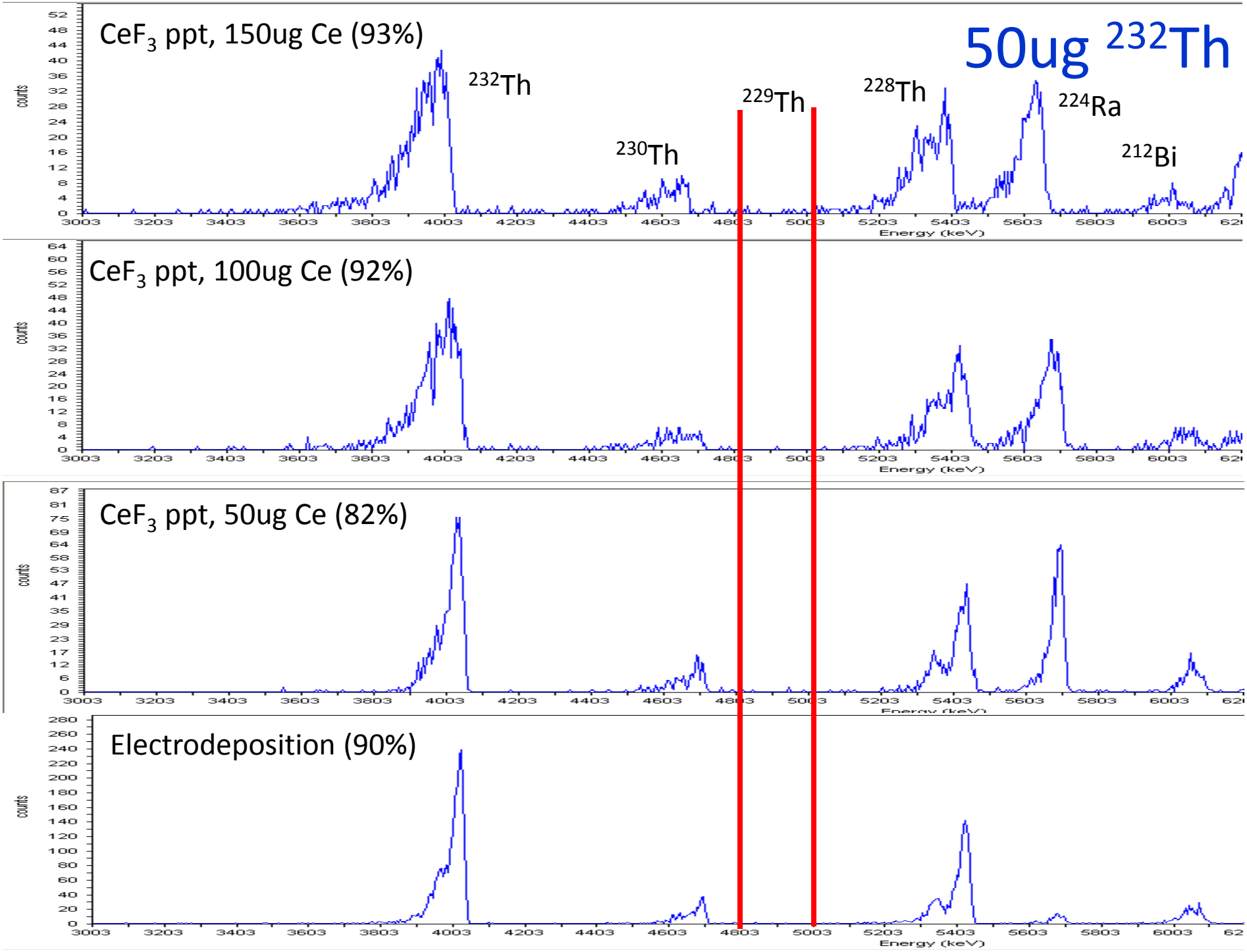

CeF3 ppt, 150ug Ce (100%)
238U
234U
235U
232U
50ug 238U
CeF3 ppt, 100ug Ce (90%)
CeF3 ppt, 50ug Ce (51%)
Electrodeposition (90%)
counts
Energy (keV)


CeF3 Microprecipitation of Th-232
30mL 4M HCl, 3mL conc. HF
% Recovery
μg Ce
0
20
40
60
80
100
0
50
100
150
200
1ug Th
5ug Th
10ug Th
25ug Th
50ug Th
100ug Th
200ug Th


CeF3 Microprecipitation of Th-232
20mL 1M HCl, 1mL conc. HF, 0.5mL 10% TiCl3
% Recovery
μg Th
0
20
40
60
80
100
0
50
100
150
200
10 ug Ce
25 ug Ce
50 ug Ce
75 ug Ce
100 ug Ce
150 ug Ce
200 ug Ce


CeF3 ppt, 150ug Ce (90%)
200ug 232Th
232Th
230Th
229Th
228Th
224Ra
212Bi
CeF3 ppt, 100ug Ce (72%)
CeF3 ppt, 50ug Ce (68%)
Electrodeposition (90%)
counts
Energy (keV)


CeF3 ppt, 150ug Ce (92%)
100ug 232Th
232Th
230Th
229Th
228Th
224Ra
212Bi
CeF3 ppt, 100ug Ce (78%)
CeF3 ppt, 50ug Ce (61%)
Electrodeposition (90%)
counts
Energy (keV)


50ug 232Th
CeF3 ppt, 150ug Ce (93%)
232Th
230Th
229Th
228Th
224Ra
212Bi
CeF3 ppt, 100ug Ce (92%)
CeF3 ppt, 50ug Ce (82%)
Electrodeposition (90%)
counts
Energy (keV)